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REACTIONS ACCOMPANYING THE FIRING OF BRICK

By

R. E. GRIM AND W. D. JOHNS, JR.

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Reactions Accompanying the Firing of Brick

by R. E. GRIM and W. D. JOHNS, JR.

Illinois State Geological Survey and Engineering Experiment Station, University of Illinois, Urbana, Illinois

Results are presented of an investigation of the reactions taking place when brick, particularly those made from fire clay containing some organic matter and sulfides, are fired. In making this investigation the differential thermal method was used. This method permitted a study of the temperature at which various reactions take place, the intensity of these reactions, and the speed with which they reach completion. The reactions for carbon are slow. The reactions for sulfur are also slow and somewhat different from those taking place when similar sulfur compounds are heated in air. The loss of (OH) lattice water from the clay mineral components is accompanied by an intense and abrupt reaction which shows slight relation to the heating rate. The structural change accompanying the loss of (OH) water fortunately causes little or no disruption of the brick.

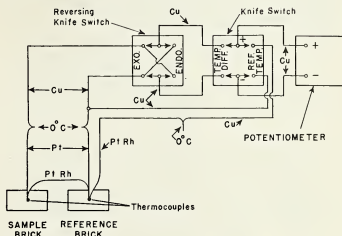


Fig. 1. Differential thermocouple circuit.

I. Introduction

THE present paper gives the results of a study of the firing of fire-clay, kaolin, and shale brick, using the differential thermal procedure which provides information regarding the thermal reactions taking place during firing. The object of the study was to obtain fundamental data that would provide a better understanding of what actually takes place within a brick when it is fired. It was hoped that this understanding would lead to the more rapid firing of ware and to a product of higher quality.

II. Procedure

The differential thermal method¹ is a means of determining the temperature at which thermal reactions take place when a

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The authors are, respectively, geologist, Illinois Geological Survey, Urbana, and special research assistant, University of Illinois, Urbana.

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¹ R. E. Grim and R. A. Rowland, "Differential Thermal Analysis of Clay Minerals and Other Hydrous Materials," *Illinois State Geol. Survey, Rept. Invest.*, No. 85, 34 pp. (1942); *Am. Mineral.*, 27, 746-61, 801-18 (1942); *Ceram. Abstracts*, 22 [6] 107 (1943).

substance is heated, the intensity of the reactions, and their rate or duration. In clay materials, reactions adsorbing heat (endothermic) may be due to dehydration, change in crystal phase, or destruction of lattice structure. Reactions giving off heat (exothermic) may be due to oxidation or the development of new crystalline phases.

The procedure used in the work reported herein is illustrated in Fig. 1. One junction of a difference thermocouple is embedded in the center of a dried unfired brick, called the test brick. The other junction is embedded in the center of a fired brick of the same kind, hereinafter called the reference brick. The two bricks and the thermocouple were placed in a large electric furnace and fired at a uniform heating rate. The two heating rates used in the experiments were (1) increasing the furnace temperature 100° C. per hour, and (2) increasing the furnace temperature 50° C. per hour. In some experiments the temperature of the furnace was held constant after it reached a given temperature, e.g., 800° C. The difference thermocouple was attached to a potentiometer which permitted a determination of the difference between the temperature at the center of the reference brick and that at the center of the test brick at any time. Any such difference should be due wholly to reactions taking place in the test brick.

Another thermocouple was placed in the furnace and attached to the potentiometer in order to determine the actual furnace temperature at any given time. As shown in Fig. 1, the thermocouple circuits were arranged so that the actual temperature at the center of each brick could be measured, as well as the difference between them.

Chemical analyses were made to determine the amount of carbon and sulfur remaining in the center of some of the brick after being fired to various temperatures.

III. Brick Used

Brick made from three different types of material were used in the experiments:

(1) Fire-clay brick were made by dry pressing an underclay of Pennsylvanian age from Grundy County, Illinois. The fire clay was composed of about 50% kaolinite, 20% illite, and 30% nonclay mineral material. Quartz in grains of silt and fine-sand size is the dominant nonclay mineral. A few per cent of organic material and sulfides, probably substantially all iron sulfide, are also present. Brick pressed from fire clay, ground to two different size grades, were used. Sieve analysis of the ground clay used are given in Table I.

(2) Kaolin brick were made by dry pressing a kaolin of Cretaceous (?) age from Union County, Illinois. Except for a very small amount of organic material (<1%) this kaolin is substantially pure kaolinite of very fine grain size. Prior to dry pressing, the kaolin was ground to approximately the grain size of the coarse grind shown in Table I.

Table I. Particle Size Analysis of Ground Fire Clay

| Screen size | Amount retained on sieve (%) | |
|-------------|------------------------------|------------|
| | Coarse grind | Fine grind |
| 6 | Trace | |
| 12 | 18.9 | |
| 20 | 29.9 | Trace |
| 30 | 12.5 | 2.0 |
| 40 | 8.6 | 3.5 |
| 50 | 6.5 | 6.6 |
| 70 | 5.1 | 7.9 |
| 100 | 4.1 | 11.7 |
| 140 | 3.7 | 10.1 |
| 200 | 2.9 | 9.4 |
| 270 | 1.4 | 6.3 |
| Pan | 6.4 | 42.5 |

(3) Shale brick were made by stiff mud extrusion from a shale of Pennsylvanian age from Vermilion County, Illinois. The shale is composed of about 70% clay mineral which is mostly illite. Some chloritic mica clay mineral and perhaps some other mica clay minerals are also present. The 30% nonclay mineral is mostly grains of quartz of silt size. A very small amount of organic material ($\pm 1\%$) and possibly a trace of sulfide are present.

IV. Experimental Data and Results

The differences between the temperatures at the centers of the reference and the test brick are plotted against the actual temperature of the center of the reference brick (Figs. 2, 3, and 4). Deflections of the curves downward indicate that the temperature at the center of the test brick is lower than that

of the reference brick and, therefore, that an endothermic reaction is taking place in the test brick. Deflections upward indicate that the temperature at the center of the test brick is the higher and, therefore, that an exothermic reaction is taking place in the test brick. Thus at point *a* on curve (A) of Fig. 2, the center of the test fire-clay brick is at a temperature 27°C. below that of the center of the reference brick when the center of the reference brick is at 200°C. At point *b* the temperature at the center of the test brick is 17°C. greater than that of the center of the reference brick when it is at 400°C.

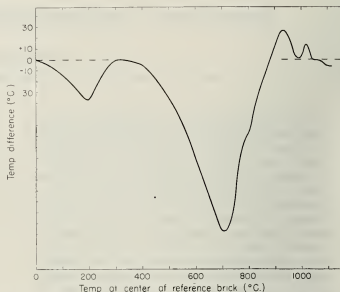


Fig. 3. Differential thermal curve for fire-clay brick fired at increase of 100°C. per hour.

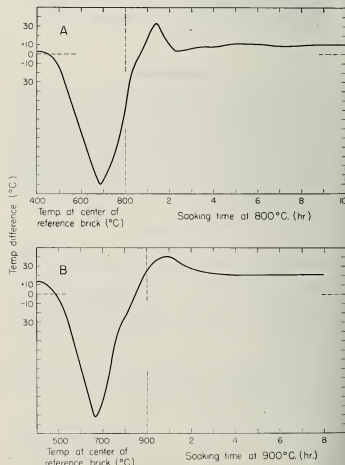


Fig. 4. Differential thermal curves for fire-clay brick fired to (A) 800°C. and (B) 900°C. at increase of 100°C. per hour, then soaked at top temperatures.

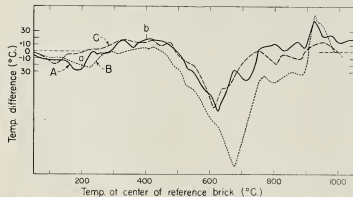


Fig. 2. Differential thermal curves for (A) fire-clay brick, (B) kaolin brick and (C) shale brick. Rate of temperature increase for all three brick was 50°C. per hour.

The difference temperatures are plotted against the temperature of the center of the reference brick, rather than the actual furnace temperature, to eliminate most of the effect of temperature lag between the center of the brick and the furnace. The effect probably cannot be eliminated completely because of possible variations in heat conductivity between the reference and the test brick and because of possible thermal reactions which may take place in the reference brick. It is believed that these effects are too small to alter the results significantly.

Figure 2 presents differential thermal curves for (A) fire-clay brick, (B) kaolin brick, and (C) shale brick all fired at a rate of temperature increase of 50°C. per hour. Curve (A) for the fire-clay brick shows an endothermic reaction up to about 200°C. due to the loss of adsorbed water which was not lost in the drying of the brick. An exothermic reaction beginning at about 275°C. causes the center of the test brick to be 5° to 18°C. hotter than that of the reference brick. This exothermic reaction cannot be interpreted in detail but it is undoubtedly due to the oxidation of organic and sulfide compounds with the former beginning first.

The exothermic reaction continues to about 450°C., when an endothermic reaction begins which continues until a temperature of 750°C. is reached in the reference brick. Above 750°C. there is again an exothermic reaction. The endothermic depression between 450° and 750°C. is due to the loss of (OH) water from the crystal structure of the clay mineral and a change in crystalline phase of the quartz from the α to β form. Actually the situation within the brick between 275°C. and about 800°C. for about 10 hours is that of slow, more or less continuous oxidation of the organic material and sulfur, producing an exothermic reaction. This reaction depends on the penetration of oxygen into the brick and on the escape of the oxidation-product gases. The reaction is slow. The endothermic reaction due to loss of (OH) water is intense and very rapid, breaking through the exothermic reaction and offsetting it for an interval of about 6 hours between 450° and 750°C.

The loss of (OH) water from the illite and kaolinite components of the brick begins at about 450°C. and is completed in about 3½ hours. During this time the temperature of the center of the reference brick has gone from 450° to 625°C. while the center of the test brick has gone from 465° to only 545°C. That is, when the reaction is completed there is a lag of 80°C. between the center of the test and that of the reference brick. After the reaction is completed the temperature in the test brick goes up to that of the reference brick in about 2½ hours, so that when the reference brick is at 750°C. the test brick has the same temperature. The loss of (OH) water from this brick corresponds to a weight loss of about 8%.

The irregularities in the curve between 500° and 750°C. cannot be explained in detail. They are undoubtedly due to the fact that the brick is made up of a mixture of clay minerals and quartz all of which show endothermic reactions in this temperature interval but not exactly at the same temperature or at the same rate.

From 750°C. until the end of the run at 1050°C. the center of the test brick is at a higher temperature than the reference brick, due to a series of exothermic reactions. Again the curve cannot be interpreted in detail, but the reactions between about 750° and 850° to 900°C. are due to the oxidation of sulfur compounds, and possibly also some organic material, which remain at even this high temperature. The portion above about 850° to 900°C. results from exothermic reactions due to the formation of new crystalline phases; for example, the exothermic peak just short of 925°C. probably corresponds to the formation of mullite from the kaolinite. In this temperature interval, from 850° or 900° to 1050°C., there would also be some endothermic reactions of relatively low intensity due to the breakdown of clay mineral structures,² which would add to the irregularities of the curve.

Curve (B) in Fig. 2, for the kaolin brick, shows an initial endothermic peak up to about 225°C. due to the loss of adsorbed water not previously removed in drying. For an interval of about 2 hours between about 350° and 450°C. there is a slight exothermic reaction due to the oxidation of the very small amount of organic material which the brick contains.

Beginning at about 450°C. there is an intense endothermic reaction, due to loss of (OH) water from the crystal structure of the kaolinite, which continues for 4½ hours. In this time interval the reference brick has reached a temperature of 675°C. whereas the temperature of the center of the test brick has reached only 505°C. In other words there is a temperature lag of 170°C. due to the endothermic reaction. The loss of (OH) water from this brick is accompanied by a loss of about 13% in weight.

About 2 hours is required after the completion of the endothermic (OH) reaction for the test brick to again approach the temperature of the reference brick. The center of the test brick does not quite regain the temperature of the center of the reference brick, as shown by the way the curve continues below the base line between about 775° and 900°C. There is probably no thermal reaction of any kind in this temperature interval. The lower temperature of the center of the test brick probably is due to a difference in rate of heat transfer through the two brick.

The endothermic peak for this brick does not show the irregularities in the similar peak for the fire-clay brick, because the kaolin brick is composed of substantially pure kaolinite.

The relatively intense exothermic reaction from 900°C. until the end of the firing is due to the formation of a new crystalline phase, mullite, from the kaolinite.³

Curve (C), for the shale brick, shows a slight initial endothermic peak due to loss of adsorbed water and a subsequent moderate exothermic reaction continuing from about 200° to 475°C. The exothermic reaction corresponds to the oxidation of a small amount of organic material.

The endothermic peak for the loss of (OH) water begins at about 475°C. The reaction is completed in about 2½ hours, at which time the reference brick has reached a temperature of about 615°C. and the test brick has a center temperature of only 530°C. The temperature of the test brick equals that of the reference brick in about 2½ hours, so that at about 750°C. they have the same temperature. The loss of (OH) water from the test brick corresponds to a weight loss of about 4%. The irregularities in the endothermic depression between about 475° and 750°C. cannot be explained in detail, but are undoubtedly the result of a series of endothermic reactions of slightly different intensities and rates caused by the combination of minerals composing the shale.

The irregularities in the curve between 750° and 900°C. are probably due to a combination of endothermic reactions resulting from destruction of the clay mineral structures, to a difference in rate of heat penetration through the test and the reference brick, and possibly also to slight exothermic oxidation reactions.

The exothermic peak above 900°C. corresponds to the formation of new crystalline phases from the clay minerals and is part of the vitrification process.

Perhaps the most significant result shown by the curves is the great speed of the reaction of the loss of (OH) water from the structure of the clay minerals. These data and the thermal curves of pure clay minerals⁴ all suggest that actually this water comes out as soon as the proper temperature is attained, and that, unlike the oxidation reactions for organic material

² R. E. Grim and W. F. Bradley, "Effect of Heat on the Clay Minerals Illite and Montmorillonite," *J. Am. Ceram. Soc.*, **23** [8] 242-48 (1940).

³ J. E. Comeforo, R. B. Fischer, and W. F. Bradley, "Multitization of Kaolinite," *J. Am. Ceram. Soc.*, **31** [9] 254-59 (1948).

and sulfur, which are slow, the endothermic reaction for loss of (OH) water cannot be slowed down. All that can be done is to reduce the rate of heat penetration into the brick and thereby retard the dehydration temperature wave which moves through a given brick, i.e., increase the temperature gradient within the brick. Austin⁴ has pointed out that stresses within a brick increase as the temperature gradient in it increase, and that, in general, optimum firing conditions require as low a gradient as possible. In no case were any of the fired brick in this investigation ruptured in any way by the rapid firing rate. It is probably better, therefore, to have a rapid heating rate to reduce the temperature gradient through the brick since the rapid loss of (OH) water, even though it may mean a weight loss of as much as 13%, does not rupture the brick.

The loss of (OH) water is an intense endothermic reaction, particularly in the case of kaolinite clays; much heat energy is adsorbed in the reaction. This means that an unusually large amount of heat must be supplied during the temperature interval of about 450° to 650°C. to carry through the dehydration reaction at a rapid rate. Stated another way, if the rate of heat input is kept the same, the huge amount of heat taken up in the reaction will slow down the dehydration wave through the brick and cause a very large temperature gradient in the brick.

The huge weight loss with the loss of (OH) water is not necessarily accompanied by shrinkage or rupture of the brick. The loss of (OH) water from the lattice of the clay minerals, illite, montmorillonite,² and probably kaolinite,³ is not accompanied by a complete destruction of the lattice or a shrinkage of the lattice structure. It is a fortunate fundamental fact, perhaps not well appreciated by those who burn brick, that the changes in the structure of the clay minerals at the time of their dehydration are of a kind not likely to disrupt the brick. Therefore, as far as the dehydration reaction alone is concerned, it is probably best to fire through the temperature range as rapidly as possible within reasonable limits.

The loss of (OH) water from the clay minerals is accompanied by the production of considerable high-temperature water vapor moving out through the brick. Oxidation reactions within the brick are dependent on the presence of oxygen which must somehow get through the brick. It seems probable that the escaping water vapor may hinder the entrance of oxygen, so that while this dehydration reaction is taking place the rate of oxidation is reduced. At the temperatures involved it seems that any oxidizing effect due to the dissociation of the steam would be insignificant. It will be shown later that the factor which controls the permissible speed of firing the brick is frequently the time required to oxidize the organic material and the sulfur compounds. Therefore, on this basis, it seems best to go through the dehydration reaction as rapidly as possible in order to reduce for only the shortest period of time the rate of oxidation.

Figure 3 presents a curve for the burning of a fire-clay brick similar to curve (A) of Fig. 2, except that the rate of temperature increase was 100°C. per hour. With the more rapid heating rate there is considerable overlap of reactions resulting from separate components, so that a smoother curve is produced.

The curve shows an initial endothermic reaction due to the loss of adsorbed water. The temperature difference between the center of the test brick and that of the reference brick is greater than it was during the same temperature interval with the slower firing rate (curve A, Fig. 2), because at any given instant during the completion of this endothermic reaction the reference brick is relatively hotter and hence the difference temperature is greater.

The heating rate is so rapid that the furnace reaches the temperature starting the endothermic reaction, due to loss of

(OH) water, so soon after the start of the exothermic reaction, due to the oxidation of the organic material and sulfide compounds, that the exothermic reaction is not definitely shown by the curve.

The loss of (OH) water begins when the center of the brick is about 400°C. and is completed in about 3 hours when the reference brick has reached 700°C. During this 3-hour interval the temperature of the center of the test brick has increased from 400°C. to about 545°C. The very great temperature difference of 155°C. at the time of completion of this reaction again is to be expected because of the rapid heating rate of the furnace and the speed of the reaction itself.

It is interesting to note that with a heating rate of 50°C. per hour, 3½ hours were required to remove the (OH) water, and that doubling the heating rate to 100°C. reduced the time required by only ½ hour, that is to 3 hours. Further, with both heating rates, the temperatures at the centers of the test brick at the time of completion of this reaction were the same, namely, 545°C. These data seem to be in accord with the conclusion just arrived at, that the (OH) water comes out of the brick as quickly as the brick is heated to a given temperature, and the process cannot be slowed down. All that can be done is to slow down the rate of movement of the wave of dehydration as it moves through the brick by increasing the temperature gradient in the brick. When the temperature at a given point in the brick reaches the dehydration temperature, that point loses its water almost instantly. The controlling factor for the rate of the movement of the dehydration wave is the rate of heat penetration into the brick versus the large latent heat necessary for the reaction.

The center of the test brick regains the temperature of the reference brick in about 2 hours at a temperature of about 875°C. Unlike the reaction for loss of (OH) water, the exothermic reactions due to the oxidation of organic and sulfide materials are slow and have not been completed by the time the temperature of 875°C. has been reached. The part of the curve between 875° and about 1050°C. shows two distinct exothermic reactions, which are due undoubtedly to the formation of new crystalline phases and to the elimination of organic and sulfur compounds. The latter reactions probably are mainly in the lower part of this temperature interval.

Figure 4 presents two curves for fire-clay brick of the same type as curve (A) in Fig. 2 and the curve in Fig. 3. One was heated at a rate of 100°C. per hour until the temperature at the center of the reference brick was 800°C. (curve A); the furnace was maintained at this temperature for 10 hours. The other was fired in the same manner until the temperature at the center of the reference brick was 900°C. (curve B), and this temperature was maintained for 8 hours. The parts of the curves below 400°C. are not presented since they add nothing to the data already considered.

In both brick the reaction for loss of (OH) water begins when the reference brick is at about 400°C. Because of the exothermic reaction taking place in the test brick its temperature is slightly higher at this point. The curves are substantially the same until about 800°C. is reached, which provides a good check of the reliability of the method.

In the case of the brick soaked at 800°C., the curve shows a fairly large exothermic reaction immediately after the endothermic reaction for loss of (OH) water, and then a very moderate exothermic reaction which continues until the end of the soaking period. The exothermic effect is probably due to oxidation. It appears that immediately after the loss of (OH) water there is a relatively large amount of oxidation followed by a slow continuing oxidation reaction. It is suggested that the relatively large exothermic peak is a consequence of the retardation of the oxidation during the escape of (OH) water in the interval just preceding.

In the case of the brick soaked at 900°C., the exothermic reaction immediately following the loss of (OH) water is relatively larger than that shown by the brick soaked at 800°C., which is to be expected since oxidation reactions would be

⁴ J. B. Austin, "The Physics and Chemistry of Firing Ceramic Ware," *Bull. Am. Ceram. Soc.*, 14 [5] 157-65 (1935).

more intense at the higher temperatures. Again the data seem to indicate that there is an actual retardation of the oxidation during the interval when (OH) water is escaping. If this is true, it is a further reason for the desirability of carrying the brick through the (OH) dehydration period as rapidly as possible. On the completion of the relatively intense exothermic peak, the temperature difference levels off and the test brick maintains a temperature of about 20°C. above that of the reference brick until the end of the soaking period. This exothermic reaction is probably due mostly to exothermic vitrification reactions with perhaps some slight oxidation at the beginning of the interval.

Table II represents determinations of the amounts of organic carbon and of sulfur in the test brick and the amounts remaining in the center of the brick after it has been fired to various temperatures.

Data⁵ obtained from differential thermal analyses of pure iron sulfides indicate that the mineral breaks down from an exothermic reaction due to the oxidation of the sulfur beginning at about 400°C. Plankenhorn⁶ has shown that when a small amount of sulfide is embedded in a large amount of clay in a brick, the elimination of the sulfur is much more complex

than would be anticipated from data for the oxidation of pure sulfides. The data obtained in the present study concur with Plankenhorn's conclusions.

The analytical data in Table II show clearly that about 40% of the total sulfur is driven out of the brick easily by the time the firing temperature reaches about 500°C. The remainder comes out slowly even at temperatures as high as 800°C. Increasing the temperature from 800° to 900°C. greatly speeds up the removal of the sulfur compounds. No attempt was made to identify the sulfur compounds remaining in the brick at relatively high temperatures.

Removal of organic carbon, like the removal of sulfur, involves relatively slow oxidation reactions, and some carbon persists to high temperatures with rapid heating rates. Also, the rate of removal of the carbon is greater as the temperature increases.

It is clear that carbon and sulfur can easily persist in a brick during firing into the vitrification temperature range (about 850° to 900°C.). The explanation of bloated burned clay is probably to be found in this persistence of carbon and sulfur to relatively elevated temperatures.

The most rapid removal of carbon and sulfur is attained by burning at the highest temperatures that can safely be reached without beginning the processes of vitrification. If the temperature is carried into the vitrification range before the carbon and the sulfur are removed, bloating may result. The persistence of gray-to-black central cores in fired brick is undoubtedly due to either carbon or sulfur compound remaining until high temperatures. Sulfur compounds, more often than carbon compounds, are the cause since the sulfur compounds are more persistent.

The data presented in Table II show the great effect of fineness of grain on the removal of sulfur and organic carbon compounds during burning. The time necessary to remove these constituents, as well as the temperature required to remove them completely, is reduced by finer grinding. This, of course, is to be expected because, other things being equal, the rate of oxidation of a given material increases as the particle size becomes smaller.

V. Conclusions

The data presented herein, derived from differential thermal studies of the firing of brick, show that the loss of (OH) water usually begins somewhat below 500°C. and is accompanied by an intense endothermic reaction. The reaction takes place abruptly as soon as any portion of the brick reaches the proper temperature. The only way this reaction can be slowed down is to reduce the rate of heat penetration through the brick. The loss of (OH) water is accomplished without complete destruction or shrinkage of the clay mineral lattice and, as a consequence, the brick does not necessarily rupture or even crack when the (OH) water is driven off with extreme rapidity. The evidence suggests that oxidation reactions are retarded during the interval when (OH) water is coming out of the brick.

Organic compounds and sulfur compounds in a brick are oxidized and eliminated slowly, accompanied by exothermic reactions. Such compounds may persist to temperatures above 800°C. when the heating rate is relatively rapid. They, particularly sulfur compounds, are generally responsible for the dark cores found on firing some brick. The removal of these compounds appears to be retarded during the interval when (OH) water is lost. It is expedited by increasing the temperature in ranges above the dehydration interval. Removal of the compounds should be completed before vitrification temperatures are reached if bloating is not desired. Decreasing particle size by finer grinding reduces the time and temperature necessary for complete removal of the carbon and sulfur compounds.

Optimum firing conditions for brick of the types studied seem to require as rapid a rise in temperature as possible to a point just below the vitrification temperature and main-

Table II. Determination of Carbon and Sulfur

| Firing conditions | Sulfur as SO ₂ (%) | Organic carbon (%) | Width of core (in.) | Description of fired brick |
|--|-------------------------------|--------------------|---------------------|--|
| Coarse grind | | | | |
| Unfired | 2.07 | 1.45 | | |
| Soaked at 550°C. for 4 hr.* | 1.30 | 0.74 | 2.0 | Core dark gray |
| Soaked at 800°C. for 4 hr.* | n.d. | n.d. | 1.2 | Core dark gray |
| Soaked at 800°C. for 8 hr.* | n.d. | n.d. | 1.0 | Core dark gray |
| Soaked at 800°C. for 12 hr.* | 1.07 | 0.60 | 0.7 | Core light gray |
| Soaked at 800°C. for 20 hr.* | n.d. | n.d. | None | |
| Heated to 880°C. in 5 hr. | 1.07 | 0.59 | 1.75 | Core black |
| Soaked at 900°C. for 4 hr.* | n.d. | n.d. | 1.0 | Core light gray |
| Soaked at 900°C. for 8 hr.* | 0.50 | None | 0.7 | Core very light gray |
| Soaked at 950°C. for 4 hr.* | n.d. | n.d. | 1.2 | Core pink, trace of gray |
| Heated to 1000°C. in 7½ hr.* | n.d. | n.d. | 2.0 | Core black, trace of pink |
| Fine grind | | | | |
| Unfired | 2.52 | 0.73 | | |
| Soaked at 500°C. for 4 hr.* | 1.54 | n.d. | 1.6 | Core medium gray |
| Soaked at 800°C. for 8 hr.* | n.d. | n.d. | 0.8 | Core light gray, pink at edge and outside of brick |
| Soaked at 800°C. for 10 hr.* | 0.29 | None | None | Outside pink |
| Soaked at 800°C. for 12 hr.* | n.d. | n.d. | None | Outside pink |
| Heated to 955°C. in 7½ hr.* | n.d. | n.d. | 2.0 | Core light pinkish gray, outside pink |
| Heated to 800°C., soaked 12 hr., then heated to 1150°C.* | n.d. | n.d. | None | Entire brick homogeneous |

* Rate of heating to reach soaking temperature was 100°C. per hour.
n.d. = not determined.

⁵ R. E. Grim and R. A. Rowland, "Differential Thermal Analysis of Clays and Shales, a Control and Prospecting Method," *J. Am. Ceram. Soc.*, 27 [3] 65-76 (1944).

⁶ W. J. Plankenhorn, "An X-ray Study of the Decomposition of Iron Pyrite in Clay." Presented at the Fifty-First Annual Meeting, The American Ceramic Society, Cincinnati, Ohio, April 26, 1949 (Structural Clay Products Division, No. 9).

nance of this temperature until the carbon and sulfur are removed. Slow firing through the low temperature range is not necessary and may even be detrimental to the quality of the ware.

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